

CLAIMS:

1. A process for the preparation of 5,5'-bi-1H-tetrazolediammonium salts, wherein oxalimidic acid  
5 dihydrazide is reacted with sodium nitrite in the presence of an acidic substance while maintaining the pH of the reaction solution in a range of from 4 to 6 to form 5,5'-bi-1H-tetrazole through the formation of an azide thereof, and the 5,5'-bi-1H-tetrazole is converted into 5,5'-bi-1H-tetrazoledisodium salt by the addition of sodium  
10 hydroxide, the 5,5'-bi-1H-tetrazoledisodium salt is further reacted with ammonium chloride or an aqueous solution thereof, and a formed ammonium salt is recovered as sparingly soluble crystals.
- 15 2. A preparation process according to claim 1, wherein there is added a weakly acidic compound having a pKa of 3 to 5, such as formic acid, acetic acid, propionic acid, octanoic acid or citric acid.
- 20 3. A preparation process according to claim 1, wherein an aqueous solution of sodium nitrite is dropwisely added at -10 to 30°C, an azide thereof is formed and a cyclization reaction is conducted at 10 to 70°C for 1 to 7 hours.
- 25 4. A preparation process according to claim 1, wherein an aqueous solution of sodium hydroxide is added to said reaction solution, and the reaction is conducted at 20 to 90°C for 1 to 5 hours to synthesize a 5,5'-bi-1H-tetrazoledisodium salt.
- 30 5. A preparation process according to claim 1, wherein ammonium chloride or an aqueous solution thereof is added to said reaction solution at 30 to 90°C, and the reaction is conducted at 50 to 90°C for 1 to 3 hours to synthesize a 5,5'-bi-1H-tetrazoledisodium salt.
- 35 6. A preparation process according to claim 1, wherein a weakly acidic compound is so added that a molar

ratio (B/A) of the weakly acidic compound (B) to the oxaldiimidic acid dihydrazide (A) is from 2.0 to 4.0.

7. A preparation process according to claim 1, wherein the sodium nitrite is so added that a molar ratio (C/A) of the sodium nitrite (B) to the oxaldiimidic acid dihydrazide (A) is from 2.0 to 4.0.

8. A preparation process according to claim 1, wherein the sodium hydroxide is so added that a molar ratio (D/A) of the sodium hydroxide (B) to the oxaldiimidic acid dihydrazide (A) is from 2.0 to 3.5.

9. A preparation process according to claim 1, wherein the ammonium chloride is so added that a molar ratio (E/A) of the ammonium chloride (B) to the oxaldiimidic acid dihydrazide (A) is from 2.0 to 3.5.

10. A process for the preparation of oxaldiimidic acid dihydrazide from dicyan and hydrazine hydrate of an amount larger than a stoichiometric ratio thereof to the dicyan.

11. A preparation process according to claim 10, wherein the reaction is conducted at  $-10$  to  $50^{\circ}\text{C}$  for 2 to 30 hours and, after the reaction, the precipitated crystals are separated.

12. A preparation process according to claim 10, wherein the reaction is conducted at a molar ratio (G/F) of the hydrazine hydrate (G) to the dicyan (F) of from 2.5 to 3.5.

13. A preparation process according to claim 10, wherein a polar solvent such as water or alcohol, or a mixed solvent thereof is used as the reaction solvent.

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